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RRES Remediation Program

Risk Reduction and Environmental Stewardship—Remediation Program

Standard Operating Procedure

for # Routine Validation of High Explosives Data

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Revision Log

Revision No.	*Effective Date*	*Prepared By*	*Description of Changes*	*Affected Pages*
R0	05/02/00	Bart Vanden Plas	Initial procedure.	All
R1	05/23/03	Karen Schultz-Paige	Rewritten to streamline and update process	All

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Routine Validation of High Explosives Data

1.0 PURPOSE

1.1 This standard operating procedure (SOP) represents the minimum standards for evaluating routine high explosives (HE) analytical data. These data can be generated for the Los Alamos National Laboratory (LANL), Risk Reduction and Environmental Stewardship—Remediation (RRES-R) Program, using SW-846 Method 8330 under the current statement of work (SOW) for analytical services (LANL 2001). The evaluation of data by this procedure is not specific to a particular data use, although this procedure may be used to develop focused data-validation requirements specific to a particular data use.

1.2 Implementation of this procedure will result in a tabulation of data compliances and noncompliances identified relative to expectations for data quality based on national guidelines for data review (U.S. Environmental Protection Agency [EPA] 1999, 66649). Data noncompliance is noted through the application of qualifiers (Attachment A) and reason codes (Attachment B) to the reported results. Because the acceptance criteria used for this procedure are not based on site-specific acceptance criteria, the results of this validation procedure are intended to be used as general indicators of data quality and should not be construed as a definitive identification of data usability.

1.3 Nothing in this SOP precludes the validator from going beyond the minimum requirements specified in this SOP. In order to address data quality issues in a data package, the validator may assign qualifiers based on his or her professional judgment. Implementation of this procedure may also be followed by a more focused and data use-specific evaluation of data, especially if implementation of this SOP indicates that the data may contain technical deficiencies. The validator will note any need for a more focused validation on the Data-Validation Cover Sheet (Attachment C). The validator will use the VOC Data-Validation Checklist (Attachment D) to record the specific validation steps conducted.

2.0 SCOPE

2.1 All **RRES-R Personnel** shall implement this mandatory SOP who evaluate routine HE analytical data for the RRES-R Program.

2.2 **Subcontractors** performing work under the RRES-R Program's quality program shall follow this SOP.

OR

2.3 **Subcontractors** may use the subcontractor's procedure, as long as the substitute meets the requirements prescribed by the RRES-R Program Quality Management Plan, and the RRES-R Program's Quality Program Project Leader (QPPL) and an RRES-R Program technical staff person approve the procedure before the subcontractor begins the designated activity.

3.0 TRAINING

3.1 **RRES-R Personnel** shall train to and use the current version of this SOP; contact the author if the SOP text is unclear.

3.2 **RRES-R Personnel** using this SOP shall document training in the RRES-R training database located at http://erinternal.lanl.gov/Training/login.asp in accordance with QP-2.2.

3.3 The responsible **supervisor** shall monitor the proper implementation of this procedure and ensure that the appropriate personnel complete all applicable training assignments.

3.4 All **data validators** implementing this SOP shall possess a minimum of a bachelors degree in chemistry or one of the physical sciences AND either two years experience in generating analytical data in an environmental analytical laboratory or two years data-validation experience.

3.5 **Validators** who are new to this SOP shall work under the direct supervision of an experienced RRES-R Program validator (who is trained to this SOP). An experienced RRES-R Program validator shall review and sign new validators' work until 10 data-record packages for this data-validation SOP are satisfactorily validated.

3.6 RRES-R Program **validators** shall demonstrate familiarity with the U.S. Environmental Protection Agency (EPA) national functional guidelines for data review.

4.0 DEFINITIONS

4.1 *Analyte*—Element, nuclide, or ion that a chemical analysis seeks to identify and/or quantify; the chemical constituent of interest.

4.2 *Continuing calibration verification (CCV)*—Check standards used to determine if the instrument response to analyte concentration is within acceptable bounds relative to the initial calibration. A CCV is performed every 12 h of operation or (for inorganics and high explosive [HEs]) every ten injections (samples and/or quality control [QC] samples); whichever is more frequent, thus verifying the satisfactory performance of an

instrument. The continuing calibration 12-h period assumes that the instrument has not been shut down since the initial calibration.

4.3 *Data validator*—Person who has met the minimum standards of training established by the RRES-R Program for data validation and who performs data validation on behalf of the RRES-R Program (hereinafter referred to as the "validator").

4.4 *Detect (inorganic and organic)*—Sample result above the method detection limit (MDL) reported by the contract analytical laboratory. The contract laboratory reports the concentration of the analyte in the sample.

4.5 *Form 1*—Organic analysis data sheet for each individual sample that includes the sample information needed to identify the sample and the analytical results for the sample. See the SOW for analytical services (RFP No. 9-XS1-Q4257) for a more complete definition.

4.6 *Holding time*—Maximum elapse of time that a sample can be stored without unacceptable changes in analyte concentrations. Holding times apply under prescribed conditions, and deviations from these conditions may affect the holding time. Extraction holding time refers to the time lapse from sample collection to sample preparation; analytical holding time refers to the time lapse between sample preparation and analysis.

4.7 *Initial calibration*—Process used to establish the relationship between instrument response and analyte concentration at several analyte-concentration values to demonstrate that an instrument is capable of acceptable analytical performance.

4.8 *Laboratory control sample (LCS)*—Known matrix that has been spiked with compound(s) representative of the target analytes. The LCS is used to document laboratory performance. The acceptance criteria for LCSs are method specific.

4.9 *Laboratory duplicate sample*—Portions of a sample taken from the same sample container, prepared for analysis and analyzed independently but under identical conditions; used to assess or demonstrate acceptable laboratory method precision at the time of analysis. Each duplicate sample is equally representative of the original material. Duplicate analyses also are performed to generate data, to determine the long-term precision of an analytical method on various matrices.

4.10 *Laboratory qualifier (or laboratory flag)*—Codes applied to the data by the contract analytical laboratory to indicate, on a gross scale, a verifiable or potential data deficiency. These flags are applied using the EPA contract laboratory program (CLP) guidelines (EPA 1994, 48639; EPA 1999, 66649).

4.11 *LANL data-validation qualifiers*—Data qualifiers defined by LANL and used in the RRES-R Program routine validation process. Attachment A lists all the data qualifiers that are applicable to all analytical suites.

4.12 *LANL data-validation reason codes*—Codes applied to the sample data by data validators who are independent of the contract laboratory that performed the sample analysis. Reason codes provide an in-depth and analysis-specific explanation for applying the qualifier along with a description of the potential impact on the data use (Attachment B). For a complete list of data qualifiers applicable to any particular analytical suite, consult the appropriate RRES-R Program SOP.

4.13 *Lower acceptance limit (LAL)*—Lowest limit that is acceptable, based on the QC criteria for a specific QC sample for a specific method. Any results lower than the LAL are qualified following this routine validation procedure.

4.14 *Matrix spike*—An aliquot of sample spiked with a known concentration of target analyte(s). Matrix-spike samples are used to measure the ability to recover prescribed analytes from a native sample matrix. Spiking typically occurs before sample preparation and analysis.

4.15 Method blank—Analyte-free matrix to which all reagents are added in the same volumes or proportions as those used in the environmental sample processing and which is prepared and analyzed in the same manner as the corresponding environmental samples. A method blank is used to assess the potential for sample contamination during preparation and analysis.

4.16 *Method detection limit (MDL)*—Minimum concentration of a substance that can be measured and reported with known statistical confidence that the analyte concentration is greater than zero. The MDL is determined by analysis of samples of a given matrix type that contain the analyte after the sample is subjected to the usual preparation and analyses. The MDL is used to establish detection status.

4.17 *Nondetect (organics)*—Sample result that is less than the MDL. The laboratory reports nondetects as undetected at the reporting limit (RL).

4.18 *Percent difference (%D)*—Measure of deviation from the initial calibration to the continuing calibration based on the calibration factors.

4.19 *Percent recovery (%R)*—Amount of material detected in a sample (minus any amount already in the sample) divided by the amount added to the sample and expressed as a percentage.

4.20 *Percent relative standard deviation (%RSD)*—Evaluation of deviation between the concentration versus analyte response over the dynamic linear calibration range. The basic equation is %RSD = (Std dev/av) • 100.

4.21 *Reporting limit (RL)*—Lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine analytical-laboratory operating conditions. The low point on a calibration curve should reflect this reporting limit. The RL is not used to establish detection status.

4.22 *Request number (RN)*—An identifying number assigned by the RRES-R Program to a group of samples that are submitted for analysis.

4.23 *Routine data*—Data generated using analytical methods that are identified as routine methods in the current RRES-R Program SOW for analytical services.

4.24 *Routine data-validation*—Process of reviewing analytical data relative to quantitative routine acceptance criteria. The objective of routine data validation is two-fold: (1) to estimate the technical quality of the data relative to minimum national guidelines adopted by the RRES-R Program and (2) to indicate to data users the technical data quality at a general level by assigning qualifier flags to environmental data whose quality indicators do not meet acceptance criteria.

4.25 *Surrogate compound (surrogate)*—Organic chemical compound used in the analyses of organic target analytes that is similar in composition and behavior to target analytes, but is not normally found in environmental samples. Surrogates are added to every blank, sample, and spike to evaluate the efficiency with which analytes are recovered during extraction and analysis.

4.26 *Target analyte*—An element, chemical, or parameter, the concentration, mass, or magnitude of which is designed to be quantified by use of a particular test method.

4.27 *Upper acceptance limit (UAL)*—Highest limit that is acceptable, based on the QC criteria for a specific QC sample for a specific method. All results greater than the UAL are qualified following this routine validation procedure.

5.0 RESPONSIBLE PERSONNEL

The following personnel are responsible for activities identified in this procedure:

- Data Validator
- RRES-R Personnel
- Project Team Leader
- Quality Program Project Leader
- Supervisor

6.0 PROCEDURE

6.1 Preparing for Data Validation

The **data validator** shall perform all the following tasks unless otherwise noted.

1. Obtain the required current versions of the HE Data-Validation Checklist form (Attachment D) from the RRES-R Program website (http://erinternal.lanl.gov/quality/forms.htm).
2. Obtain from the Sample Management Office (SMO) all data packages that contain the sample data to be validated.
   A. Prepare a Data-Validation Cover Sheet (Attachment C) by completing the cover sheet and placing a check or other mark adjacent to the analytical suites for which this validation is being performed.
   B. If any data are rejected, check the rejected box and the project chemist will be notified immediately.

      Note: You may use a single cover sheet when validating multiple analytical suites under the same RN.

      Note: Use a separate sheet of paper to document each deficiency identified beyond the scope of this procedure, including phone conversations with the analytical laboratory concerning these deficiencies. Attach these sheets to the Data-Validation Cover Sheet.
3. Verify that the following items are present in the data package:
   - Signed LANL COC record
   - Case narrative
   - Result forms (Form 1: Organic Analysis Data Sheet: Tentatively Identified Compounds or equivalent) for each sample
   - QC forms (CLP 2A [Surrogate Recovery], 3A [Matrix Spike/Matrix Spike Duplicate Recovery], 4A [Method Blank Summary], 6A [Initial Calibration Data], 7A [Calibration Verification Summary], 8A [Internal Standard Area and RT Summary], or equivalent) for water and/or soils, as appropriate
   - Chromatograms, quantitation reports, and confirmation data for all samples and blanks

4.

IF the required documentation for the data record package is…	FOR...	THEN…
Complete,		• Go to Step 5.
Missing,	< 6 mo.	• Contact the analytical laboratory and/or SMO. • Allow 3 days for submittal. • Go to Step 5.
Missing,	= 6 mo.	• Contact the analytical laboratory and/or SMO. • Allow 10 business days for submittal. • Go to Step 5.

Note: To expedite the validation process, the validator may request that the contract laboratory forward the missing information by e-mail or fax directly to them within 24 h of notification.

5.

IF the analytical laboratory…	THEN…
Submits the documentation within the specific period of time,	• Go to Step 6.
Does not submit the documentation within the specified period of time,	• Notify the SMO for contract-compliance action.

6. Record the presence or absence (“Yes” or “No”) of each item, as appropriate, in the completeness check section of the Data-Validation Cover Sheet (Attachment C).

Note: In the Data-Validation Cover Sheet completeness check section, indicate any samples whose data are missing from the data package under comments/problems noted.

7. Photocopy the following items for use during the validation process:
   - The case narrative from the data package
   - The Form 1 that you will use during the validation process before completing the form

- Chain of custody forms

Note: Do not record data-validation qualifiers and reason codes on the original form (Form 1).

Note: The validator must submit the photocopies of the items listed in Step 7 as attachments to the completed Data-Validation Checklist. Each page of the Form 1 must be signed and dated by the validator; this signature and date must be present even if the validator accepts laboratory qualification.

6.2 Verifying the Initial Calibration

1.

IF initial calibration information…	**THEN…**
Present,	• Record "No" on line 5 of the HE Data-Validation Checklist. • Go to Step 2.
Missing,	• Record "Yes" on line 1 of the HE Data-Validation Checklist. • Contact the analytical laboratory and/or SMO to request the missing information (see Section 6.1-4). • If the laboratory is unable to provide the missing information, qualify all results as rejected (R, H16) on the individual sample Form 1. • Go to Step 2.

2.

IF the initial calibration…	**THEN…**
Has five calibration points,	• Check "No" on line 2 of the HE Data-Validation Checklist. • Go to Step 3.
Does <u>not</u> have five calibration points,	• Check "Yes" on line 2 of the HE Data-Validation Checklist. • Qualify the affected analytes as estimated (J, H7/UJ, H7) on the individual sample Form 1. • Go to Step 3.

3.

IF the initial calibration does…	**THEN…**
Have a standard at or below the reporting limit,	• Check "No" on line 3 of the HE Data-Validation Checklist. • Go to Step 4.
Not have a standard at or below the reporting limit,	• Check "Yes" on line 3 of the HE Data-Validation Checklist. • Qualify the affected analytes between the MDL and the lowest standard analyzed as estimated (J, H7c) and reject all nondetected analytes (R, H7c) on the individual sample Form 1. • Go to Step 4.

4.

IF the percent relative standard deviation (%RSD) for…	**THEN…**
Each analyte is = 20% (if the RSD is not used, then the correlation coefficient = to 0.995 is used to qualify data),	• Check "No" on line 4 the HE Data-Validation Checklist. • Go to Section 6.3, "Verifying the Continuing Calibration."
Any analyte is > 20% (or the correlation coefficient is < 0.995),	• Check "Yes" on line 4 the HE Data-Validation Checklist. • Qualify all the affected analytes as estimated (J, H7a/UJ, H7a) on the individual sample Form 1. • Go to Section 6.3, "Verifying the Continuing Calibration."

6.3 Verifying the Continuing Calibration

1.

IF the continuing calibration information is…	**THEN…**
Present and analyzed at the proper frequency,	• Record "No" on line 6 of the HE Data-Validation Checklist. • Go to Step 2.
Missing or has not analyzed at the proper frequency,	• Record "Yes" on line 5 of the HE Data-Validation Checklist. • Contact the analytical laboratory and the SMO to request the missing information (see Section 6.1-4). • If the laboratory is unable to provide the missing information, qualify all the results as rejected (R, H16) on the individual sample Form 1. • Go to Step 2.

2.

IF the percent difference (%D) for…	**THEN…**
Each analyte is = 15%,	• Check "No" on line 6 of the HE Data-Validation Checklist. • Go to Section 6.4, "Verifying the Retention Times."
Any analyte is > 15%,	• Check "Yes" on line 6 of the HE Data-Validation Checklist. • Qualify all affected analytes as estimated (J, H7a/UJ, H7a) on the individual sample Form 1. • Go to Section 6.4, "Verifying the Retention Times."

Note: The qualification of the data for any continuing calibration problem affects the samples injected both before and after the failing CCV. The validator must qualify all the samples that are affected.

6.4 Verifying the Retention Times

1.

IF…	**THEN…**
All the retention time window information is present,	• Check "No" on line 5 of the HE Data-Validation Checklist. • Go to Step 2.
Any retention time window information is missing,	• Record "Yes" on line 7 of the HE Data-Validation Checklist. • Contact the analytical laboratory and the SMO to request the missing information (see Section 6.1-4). • If the laboratory is unable to provide the missing information, qualify all results as rejected (R, H11) on the individual sample Form 1. • Go to Step 2.

2.

IF the retention times for any of the compounds have…	**THEN…**
Not shifted by more than 0.05 minutes from the initial calibration,	• Check "No" on line 8 of the HE Data-Validation Checklist. • Go to Section 6.5, "Verifying the Method-Blank Results."
Shifted by more than 0.05 minutes from the initial calibration,	• Check "Yes" on line 8 of the HE Data-Validation Checklist. • Qualify all the affected analytes as rejected (R, H11a) for all the affected samples on the individual sample Form 1. • Go to Section 6.5, "Verifying the Method-Blank Results."

6.5 Verifying the Method-Blank Results

1.

IF the method-blank information is…	**THEN…**
Present,	• Record “No” in line 9 of the HE Data-Validation Checklist. • Go to Step 2.
Missing,	• Record “Yes” on line 9 of the HE Data-Validation Checklist. • Contact the analytical laboratory and the SMO to request the missing information (see Section 6.1-4). • If the laboratory is unable to provide the missing information, qualify all the results as rejected (R, H4b) on individual sample Form 1. • Go to Step 2.

2.

IF the method blank has…	**THEN…**
No contamination,	• Check “No” on line 10 of the HE Data-Validation Checklist. • Go to Section 6.6, “Verifying the Confirmation Results.”
Contamination,	• Go to Step 3.

3.

IF the concentration of any analyte in a sample is…	**THEN…**
= 5 times the concentration of that analyte in the corresponding blank,	• Check “Yes” on line 10 of the HE Data-Validation Checklist. • Qualify all the affected analytes as undetected (U, H4) for all the affected samples on the individual sample Form 1. • Go to Section 6.6, “Verifying the Confirmation Results.”

3.

IF the concentration of any analyte in a sample is…	THEN…
> 5 times the concentration of that analyte in the corresponding blank,	• The data do not require qualification. Go to Section 6.6, "Verifying the Confirmation Results."

Note: Check the concentrations of laboratory contaminants in the diluted samples. If the concentration in the sample is within ten times the concentration of the blank when the dilution factor is taken into account, and the sample was NOT diluted for that analyte, use professional judgment to apply the qualification criteria listed in this section.

6.6 Verifying the Confirmation Results

Note: Only positive results need confirmation. The confirmation column is held to the same criteria as the primary column for all qualification criteria. Only proceed through Sections 6.2 and 6.3 to qualify data on calibration of the confirmation column if there are potential detections on the primary column.

Note: When the method code HEXPUV is requested, the laboratory must also include the UV spectra as part of the confirmation data for all positive detections. Check "n/a" on lines 12 or 13 if either type of confirmation does not apply to the data package being validated.

1.

IF the required confirmation information is…	THEN…
Present,	• Go to Step 2.
Missing,	• Check "Yes" on line 11 of the HE Data-Validation Checklist. • Contact the analytical laboratory and SMO to request the missing information (see Section 6.1-4). • If the laboratory is unable to provide the missing information, qualify all results as rejected (R, H8a) on the individual sample Form 1. • Go to Step 2.

2.

IF the analyte reported has…	THEN…
Positive confirmation and passes all QC requirements (or no confirmation is required),	• Check "No" on lines 11,12, and 13 of the HE Data-Validation Checklist. • Go to Section 6.7, "Verifying the Surrogate Recoveries."
Not been confirmed or does not pass all the QC requirements,	• Go to Step 3.

3.

IF the analyte reported was…	THEN…
Confirmed on the cyano column,	• Go to Step 4.
Not confirmed on the cyano column,	• Check "Yes" on line 12 of the HE Data-Validation Checklist. • Qualify all the affected analytes as undetected (U, H8) on the individual Form 1. • Go to Step 4.

4.

IF the UV spectrum for…	THEN…
Each positive detection confirms the reported detections,	• Check "No" on Line 13 of the HE Data-Validation Checklist. • Go to Section 6.7, "Verifying the Surrogate Recoveries."
Any positive detection does not confirm the reported detections (the lab qualified the reported result with an "X" flag),	• Check "Yes" on Line 13 of the HE Data-Validation Checklist. • Qualify all the affected positive detections as undetected (U, H8b) on the individual sample Form 1. • Go to Section 6.7, "Verifying the Surrogate Recoveries."

6.7 Verifying the Surrogate Recoveries

Note: Surrogate %R that are out of specification as a result of the sample dilution used to bring the detected analyte concentrations into the instrument

calibration range are not subject to the validation-acceptance criteria listed in Table 6.7-1.

Table 6.7-1
Valid HE Surrogates and Recovery Acceptance Ranges

Surrogate	Soil Matrix Acceptance Range (%R)	Water Matrix Acceptance Range (%R)
1,2-Dinitrobenzene	50–150	60–125
1,4-Dinitrobenzene	67–116	34–106
3,4-Dinitrotoluene	50–150	88–110
4-Nitroaniline	50–150	86–118

1.

IF the surrogate information is…	**THEN…**
Present,	• Record “No” on line 14 of the HE Data-Validation Checklist. • Go to Step 2.
Missing (only one surrogate is required),	• Check “Yes” on line 14 of the HE Data-Validation Checklist. • Contact the analytical laboratory and the SMO to request the missing information (see Section 6.1-4). • If the laboratory is unable to provide the missing information, qualify all results as rejected (R, H3f) on the individual sample Form 1 • Go to Step 2.

2.

IF…	**THEN…**
Surrogate %R for a sample is < the UAL,	• Check “No” on line 15 of the HE Data-Validation Checklist. • Go to Step 3.

2.

IF…	THEN…
Any surrogate %R for a sample is > the UAL,	• Check "Yes" on line 15 of the HE Data-Validation Checklist. • Qualify all the detected analytes as estimated with a potential positive bias (J+, H3) on the individual sample Form 1. • Go to Step 3.

3.

IF…	THEN…
No surrogate %R for a sample is < the LAL,	• Check "No" on lines 16, 17, and 18 of the HE Data-Validation Checklist. • Go to Section 6.8, "Verifying the Laboratory Control Sample Recoveries."
Any surrogate %R for a sample is < the LAL and another surrogate %R in that same sample is > the UAL,	• Check "Yes" on line 16 of the HE Data-Validation Checklist. • Qualify all affected analytes as estimated (J, H3e/UJ, H3e) on the individual sample Form 1. • Go to Section 6.8, "Verifying the Laboratory Control Sample Recoveries."
Any surrogate % R for a sample is < the LAL but = 10%,	• Check "Yes" on line 17 of the HE Data-Validation Checklist. • Qualify all detected analytes as estimated with a potential low bias (J-, H3a) and all undetected analytes estimated (UJ, H3c) on the individual sample Form 1. • Go to Section 6.8, "Verifying the Laboratory Control Sample Recoveries."

3.

IF…	THEN…
The surrogate %R in a sample is < 10%,	• Check “Yes” on line 18 of the HE Data-Validation Checklist. • Qualify all detected analytes as estimated with a potential low bias (J-, H3b) and all undetected analytes as rejected (R, H3d) on the individual sample Form 1. • Go to Section 6.8, “Verifying the Laboratory Control Sample Recoveries.”

6.8 Verifying the Laboratory Control Sample Recoveries

1.

IF the laboratory control sample (LCS) information is…	THEN…
Present,	• Record “No” on line 19 of the HE Data-Validation Checklist. • Go to Step 2.
Missing (UAL and LAL values must be present on the forms containing the LCS results),	• Check “Yes” on line 19 of the HE Data-Validation Checklist. • Contact the analytical laboratory and SMO to request the missing information (see Section 6.1-4). • Use professional judgment to qualify the affected samples if LCS information is not available upon request. • Go to Step 2.

2.

IF any LCS analyte %R is…	THEN…
< the UAL,	• Check “No” on line 20 of the HE Data-Validation Checklist. • Go to Step 3.

2.

IF any LCS analyte %R is…	THEN…
> the UAL,	• Check “Yes” on line 20 of the HE Data-Validation Checklist. • Qualify all detected analytes as estimated with a potential high bias (J+, H12d) on the individual sample Form 1. • Go Step 3

3.

IF any LCS analyte %R is…	THEN…
> the LAL,	• Check “No” on lines 21 and 22 of the HE Data-Validation Checklist. • Go to Section 6.9, “Verifying the Holding-Time Results.”
< the LAL but = 10%,	• Check “Yes” on line 21 of the HE Data-Validation Checklist. • Qualify all detected analytes as estimated with a potential low bias (J-, H12b) and all undetected analytes as estimated (UJ, H12c) on the individual sample Form 1. • Go to Section 6.9, “Verifying the Holding-Time Results.”
< 10%,	• Check “Yes” on line 22 of the HE Data-Validation Checklist. • Qualify all detected analytes as estimated with a potential low bias (J-, H12a) and all undetected analytes as rejected (R, H12a) on the individual sample Form 1. • Go to Section 6.9, “Verifying the Holding-Time Results.”

6.9 Verifying the Holding-Time Results

Table 6.11-1
Holding Time Acceptance Criteria

Sample Matrix	Extraction Holding Time (days)	Analysis Holding Time (days)
Soil	14	40
Water	7	40
The current SOW for analytical services lists applicable storage conditions.		

1.

IF…	**THEN…**
All the samples were extracted and analyzed within the holding time acceptance criteria,	• Check "No" on lines 23, 24, and 25 of the HE Data-Validation Checklist. • Go to Section 6.10, "Verifying the Dilutions."
Any samples were not extracted within the holding time acceptance criteria,	• Calculate the number of days the holding time was exceeded. • Go to Step 2.

2.

IF the extraction holding time…	**THEN…**
Did not exceed 2 times the holding time acceptance criteria,	• Check "No" on line 24 of the HE Data-Validation Checklist. • Check "Yes" on line 23 of the HE Data-Validation Checklist. • Qualify all the detected analytes as estimated with a potential low bias (J-, H9) and all the undetected analytes as estimated (UJ, H9) for all affected samples on the individual sample Form 1. • Go to Step 3.

2.

IF the extraction holding time…	**THEN…**
Exceeds 2 times the holding time acceptance criteria,	• Check “Yes” on line 24 on the HE Data-Validation Checklist. • Check “No” on line 23 on the HE Data-Validation Checklist. • Qualify all the results as rejected (R, H9a) for all the affected samples on the individual sample Form 1. • Go to Step 3.

3.

IF the analytical holding time…	**THEN…**
Did <u>not</u> exceed the holding time acceptance criteria,	• Check “No” on line 25 of the HE Data-Validation Checklist. • Go to Section 6.10, “Verifying the Dilutions.”
Exceeded the holding time acceptance criteria,	• Check “Yes” on line 25 on the HE Data-Validation Checklist. • Qualify that all the results are rejected (R, H9b) in all affected samples on the individual sample Form 1. • Go to Section 6.10, “Verifying the Dilutions.”

6.10 Verifying the Dilutions

IF the sample was…	**THEN…**
Not diluted,	• Record “No” on line 26 of the HE Data-Validation Checklist. • Go to Section 6.11, “Identifying the Obvious Quality Deficiencies.”

IF the sample was…	THEN…
Diluted, and there are no target analytes detected above the second lowest standard,	• Record “Yes” on line 26 of the HE Data-Validation Checklist. • Contact the analytical laboratory and the SMO to inform the laboratory of the contract nonconformance. • If the laboratory cannot provide proof of matrix interference that could not be removed by acceptable cleanup methods, qualify the affected samples as estimated (R, H10). • If the laboratory can provide proof-of-matrix interference that was not removed by acceptable cleanup attempts, qualify all the nondetected analytes as estimated (UJ, H10) on the individual sample Form 1. • Go to Section 6.11, “Identifying the Obvious Quality Deficiencies.”

6.11 Identifying the Obvious Quality Deficiencies

IF…	THEN…
No obvious quality deficiencies exist (outside of those covered by this SOP),	• Record “No” on line 27 of the HE Data-Validation Checklist. • Go to Section 6.12, “Assembling and Submitting the Data-Validation Record Package.”

IF…	THEN…
Any obvious/significant data quality deficiencies found during the validation process,	• Record "Yes" on line 27 of the HE Data-Validation Checklist. • Contact the analytical laboratory and SMO, if necessary, to resolve the quality issue. • Record the appropriate qualifier for the data based on the validator's best professional judgment and apply Reason Code H19. • Write up a clear description of the flagged quality issue on the Data-Validation Cover Sheet. • Go to Section 6.12, "Assembling and Submitting the Data-Validation Record Package."

6.12 Assembling and Submitting the Data-Validation Record Package

1. Complete the Data-Validation Cover Sheet by signing and dating it.
2. Assemble the following items in order:
   - Completed Data-Validation Cover Sheet
   - Completed HE Data-Validation Checklist
   - Photocopies of the completed Form 1s (on which the validator recorded the qualifier flags and reason codes)

     Note: The forms must be dated and initialed by the validator.
   - Chain of custody forms
3. Submit the Validation-Data Record Package to the FSF, in accordance with SOP-15.09, "Chain of Custody for Analytical Data Packages."

7.0 LESSONS LEARNED

7.1 Before performing work described in this SOP, RRES-R Personnel should go to the Department of Energy Lessons Learned Information Services home page, located at http://www.tis.eh.doe.gov/ll/ll.html, and/or to the LANL Lessons Learned Resources web page, located at http://www.lanl.gov/projects/lessons_learned/, and search for applicable lessons.

7.2 During work performance and/or after the completion of work activities, RRES-R Personnel, as appropriate, shall identify, document, and submit lessons learned in accordance with the LANL, Lessons Learned System located at http://www.lanl.gov/projects/lessons_learned/.

8.0 RECORDS

Although no records are submitted to the Records Processing Facility (RPF) in the course of completing this procedure, the items identified in Section 8.11 are a part of the data record package submitted to the RPF from the SMO in accordance with SOP-15.09.

9.0 REFERENCES

- EPA (US Environmental Protection Agency), "U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," Publication 9240.1-05, EPA-540/R-94/012, Office of Solid Waste and Emergency Response, Washington, DC, February 1994.
- SOP-15.09, Chain of Custody for Analytical Data Packages
- LANL (Los Alamos National Laboratory), "Environmental Restoration Project Statement of Work for Analytical Services," Los Alamos National Laboratory, Revision 2, RFP Number 9-SX1-Q4257, Los Alamos, New Mexico, July 1995.
- QP-2.2, Personnel Orientation and Training
- QP-3.2, Lessons Learned
- QP-5.7, Notebook Documentation for Environmental Restoration Technical Activities

10.0 ATTACHMENTS

The **user** of this SOP may locate all forms associated with this procedure at http://erinternal.lanl.gov/Quality/ user/forms.asp.

Attachment A: High Explosives Data-Validation Qualifier Flags, 1 page

Attachment B: High Explosives Data-Validation Reason Codes, 3 pages

Attachment C: Data-Validation Cover Sheet, 1 page

Attachment D: HE Data-Validation Checklist, 1 page

Attachment E: List of Acronyms and Abbreviations, 1 page

Attachment A: High Explosives Data-Validation Qualifier Flags

R The analyte is classified “rejected”.

J The analyte is classified “detected,” but the reported concentration value is expected to be more uncertain than usual.

J+ The analyte is classified “detected,” but the reported concentration value is expected to be more uncertain than usual with a potential positive bias.

The analyte is classified “detected,” but the reported concentration value is expected to be more uncertain than usual with a potential negative bias.

U The analyte is classified “not detected.”

UJ The analyte is classified “not detected” with an expectation that the reported result is more uncertain than usual.

Attachment B: High Explosives Data-Validation Reason Codes

Code	HE	Qualifier Nondetects	Qualifier Detects	Description	Comments
3	H3	N/A	J+	Results for the affected analytes are considered estimated and biased high (J+) because the associated surrogate recovery was above the UAL.	Code is used for detected analytes.
3a	H3a	N/A	J-	Results for the affected analytes are considered estimated and biased low (J-) because the associated surrogate recovery was less than the LAL but greater than or equal to10%.	Code is used for detected analytes.
3b	H3b	N/A	J-	Results for the affected analytes are considered estimated and biased low (J-) because the associated surrogate recovery was less than 10%.	Code is used for detected analytes.
3c	H3c	UJ	N/A	Reporting limits for the affected analytes are considered estimated (UJ) because the associated surrogate recovery was less than the LAL but greater than or equal to 10%.	Code is used for nondetected analytes.
3d	H3d	R	N/A	Reporting limits for the affected analytes are considered rejected (R) because the associated surrogate recovery was less than 10%.	Code is used for nondetected analytes.
3e	H3e	UJ	J	Reporting limits/results for the affected analytes are considered estimated (UJ)/(J) because at least one of the associated surrogate was above the UAL and one was below the LAL.	
3f	H3f	R See comments	R See comments	Required surrogate information is missing. Validation cannot proceed without this information.	Information should be requested from the laboratory, or the package should be returned to the SMO.
4	H4	N/A	U	Results for the affected analytes are considered not detected (U) because the associated sample concentration was less than or equal to 5X the amount in the method blank.	Effective dilutions must be considered for common laboratory contaminates.
4b	H4b	R See comments	R See comments	Required method-blank documentation is missing. Validation cannot proceed without this information.	Information should be requested from the laboratory, or the package should be returned to the SMO.

Code	HE	Qualifier Nondetects	Qualifier Detects	Description	Comments
7	H7	UJ	J	Results for affected analytes are considered estimated (UJ)/(J) because the associated analyte did not have a valid 5-point calibration.	Qualify only the affected analytes.
7a	H7a	UJ	J	Results/reporting limits for affected analytes are considered estimated (J)/estimated (UJ) because the associated %RSD/%D exceeded criteria in the initial or continuing calibration standards.	Qualify only the affected analytes.
7c	H7c	R	J See comments	Results for the affected analytes are considered rejected (R)/estimated (J) because the associated analyte did not have a standard at the reporting limit.	Qualify as J only those results reported as detected between the lowest standard analyzed and the MDL. Results within the valid calibration range should not be qualified.
8	H8	N/A	U	Reported analyte was not confirmed during the analysis of a second dissimilar column; and therefore, is presumed to be absent from the sample.	
8a	H8a	R See comments	R See comments	Required confirmation column analysis or UV spectrum documentation is missing. Data may not be acceptable for use.	Information should be requested from the laboratory or the package should be returned to SMO.
9	H9	UJ	J-	Results/reporting limits for affected analytes are considered estimated and biased low (J-)/estimated (UJ) because the extraction holding time was exceeded by more than 2x.	
9a	H9a	R	R	Results for the affected analytes are considered rejected (R) because the sample extraction exceeded 2X the holding time.	
9b	H9b	R	R	Results for the affected analytes are rejected (R) because the analytical holding time was exceeded.	
10	H10	See comments	See comments	Undetected results for affected analyte are considered estimated (UJ) or rejected (R) because the laboratory diluted the sample for matrix interferences.	Qualify all the results as rejected if the laboratory cannot provide proof of cleanup or matrix interferences. Qualify the nondetected results as estimated if the laboratory can provide evidence of cleanup and/or matrix interferences not subject to acceptable cleanup methods.
11	H11	R See comments	R See comments	Required retention time documentation is missing. Validation cannot proceed without this information.	Information should be requested from the laboratory or the package should be returned to the SMO.

Code	HE	Qualifier Nondetects	Qualifier Detects	Description	Comments
11a	H11a	R	R	Results for affected analytes are considered rejected (R) because the associated retention times have shifted by more than 0.05 minutes from the mid-level standard of the initial calibration.	Validator must indicate on the validation cover sheet that a focused validation is needed to determine if any false negative or false positive results were reported and to determine if the data can be used. This code is only used for the CCV.
12	H12	R See comments	R See comments	LCS documentation is missing. Validation cannot proceed without this information.	Information should be requested from the laboratory or the package should be returned to the SMO.
12a	H12a	R	J-	Results/reporting limits for the affected analytes are considered estimated and biased low (J-)/rejected (R) because the associated LCS recovery was less than 10%.	
12b	H12b	N/A	J-	Results for the affected analyte are considered estimated and biased low (J-) because the associated LCS recovery was less than the LAL but greater than or equal to 10%.	Code is for detected analytes.
12c	H12c	UJ	N/A	Reporting limits for the affected analyte are considered estimated (UJ) because the associated LCS recovery was less than the LAL but greater than or equal to 10%.	Code is for nondetected analytes.
12d	H12d	N/A	J+	Results for the affected analyte are considered estimated and biased high (J+) because the associated LCS recovery was greater than the UAL.	Code is for detected analytes.
16	H16	R See comments	R See comments	Required calibration information is missing or samples were analyzed on an expired calibration. Validation cannot proceed without this information.	Information should be requested from the laboratory, or the package should be returned to the SMO. This code should also be used if the CCVs were not analyzed at the proper frequency.
19	I19	See comments	See comments	Validator identified quality deficiencies in the reported data that require qualification. See the Data-Validation Cover Sheet for specific details.	Apply the appropriate qualifier to identify the effect of the quality deficiency on the reported data.

Attachment C: Data-Validation Cover Sheet

☐ **Rejected Data**

Section I

Request Number: _____ Validation Date: _____ Lab Code: _____

Contract Laboratory Name: _____

Validator: _____ Organization: _____

Analytical Suite (check all that apply):
☐ Volatile Organics
☐ Semivolatile Organics
☐ Organochlorine Pesticides/Polychlorinated Biphenyls
☐ High Explosives
☐ Inorganics
☐ Radiochemistry

Other (describe): _____

Section II—Completeness Check

Yes	No	n/a	(check one)	Yes	No	n/a	(check one)
☐	☐	☐	1. Chain-of-custody form(s)	☐	☐	☐	6. Raw/BSS data
☐	☐	☐	2. Case narrative	☐	☐	☐	7. Quality control forms
☐	☐	☐	3. Sample result forms	☐	☐	☐	8. Quantitation reports
☐	☐	☐	4. Sample chromatograms	☐	☐	☐	9. TICs forms
☐	☐	☐	5. Standard chromatograms	☐	☐	☐	10. TICs mass spectra

Identify any samples in the assigned Request Number that are missing:

_____ _____ _____ _____ _____ _____ _____ _____

_____ _____ _____ _____ _____ _____ _____ _____

Comments/problems noted (include information about requests for further information submitted to the contract laboratory and agreed-upon date of resolution and contract laboratory point of contact):

(Attach additional comment sheets as necessary)

Validator's signature: _____ Date: _____

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Attachment D: HE Data-Validation Checklist

Yes	No	n/a	(check one)	Assign qualifier listed below if criteria = Yes: Detected analyte	Assign qualifier listed below if criteria = Yes: Undetected analyte
☐	☐	☐	1. Initial calibration not present	R, H16	R, H16
☐	☐	☐	2. Initial calibration does not have 5 calibration points	J, H7	UJ, H7
☐	☐	☐	3. Initial calibration does not have low standard at the reporting limit	--[a], H7c	R, H7c
☐	☐	☐	4. Initial calibration analyte %RSD is >20% or R is <0.995	J, H7a	UJ, H7a
☐	☐	☐	5. CCV not present or analyzed at proper frequency	R, H16	R, H16
☐	☐	☐	6. Continuing calibration analyte %D is >15%	J, H7a	UJ, H7a
☐	☐	☐	7. RT window information is not present	R, H11	R, H11
☐	☐	☐	8. RT window for individual analytes in the CCV have shifted more than 0.05 minutes from the RT window from the initial calibration	R, H11a	R, H11a
☐	☐	☐	9. Method blank results are not reported	R, H4b	R, H4b
☐	☐	☐	10. Analyte detected in method blank and sample result for analyte =5x the amount in method blank	U, H4	N/A
☐	☐	☐	11. Confirmation information is not present for positive results	R, H8a	R, H8a
☐	☐	☐	12. Analyte reported was not confirmed on a second dissimilar column or detector	U, H8	N/A
☐	☐	☐	13. UV spectrum does not confirm the reported detection	U, H8b	N/A
☐	☐	☐	14. Surrogate information is not present	R, H3f	R, H3f
☐	☐	☐	15. Surrogate % recovery >UAL	J+, H3	N/A
☐	☐	☐	16. At least one surrogate recovery is <LAL and at least one is >UAL	J, H3e	UJ, H3e
☐	☐	☐	17. Surrogate % recovery is <LAL but =10%	J-, H3a	UJ, H3c
☐	☐	☐	18. Surrogate % recovery is <10%	J-, H3b	R, H3d
☐	☐	☐	19. LCS information is not present	R, H12	R, H12
☐	☐	☐	20. LCS % recovery is >UAL	J+, H12d	N/A
☐	☐	☐	21. LCS % recovery is <LAL but =10%	J-, H12b	UJ, H12c
☐	☐	☐	22. LCS % recovery is <10%	J-, H12a	R, H12a
☐	☐	☐	23. Sample was extracted =2x the appropriate holding time	J-, H9	UJ, H9
☐	☐	☐	24. Sample was extracted >2x the appropriate holding time	R, H9a	R, H9a
☐	☐	☐	25. Sample was analyzed outside the analytical holding time	R, H9b	R, H9b
☐	☐	☐	26. Sample was diluted inappropriately	N/A	U, H10
☐	☐	☐	27. Other obvious data quality issues identified	__, H19	__, H19

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[a] Apply the J qualifier only to data that are between the MDL and the low standard.

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Attachment E: List of Acronyms and Abbreviations

CLP	contract laboratory program
COC	chain of custody
EPA	U.S. Environmental Protection Agency
RRES-R	risk reduction and environmental stewardship—remediation
FSF	Field Support Facility
HE	high explosives
LAL	lower acceptance limit
LANL	Los Alamos National Laboratory
LCS	(contract analytical) laboratory control sample
MDL	method detection limit
n/a	not analyzed
%D	percent difference
%R	percent recovery
%RSD	percent relative standard deviation
QC	quality control
RL	reporting limit
RPF	Records Processing Facility
RT	retention time
SMO	Sample Management Office
SOP	standard operating procedure
SOW	statement of work
TIC	tentatively identified compound
UAL	upper acceptance limit